Water decolorization using UV radiation and hydrogen peroxide: a kinetic study

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Abstract Sometimes, provision of water for domiciliary consumption faces the problem of natural contamination originated by the presence of organic substances such as humic or fulvic acids. Very often, after conventional sanitary treatments this water exhibits a persistent yellowish coloration that affects its use. Moreover, these substances may act as precursors of tri-halomethanes formation during pre-disinfection with chlorine. This paper presents, with a simplified mechanistic approach, the intrinsic reaction kinetics of natural water decolorization employing UV radiation and hydrogen peroxide. The main variables for the model are: contaminant concentration expressed as TOC, hydrogen peroxide concentration and the photon absorption rate.

Keywords Water decolorization; UV radiation; hydrogen peroxide; photoreactor; reactor modeling

Introduction

Advanced Oxidation Technologies can be used for treating a large variety of pollutants in air and water. The use of UV radiation has been proposed as a suitable alternative in the past decade (Legrini *et al.*, 1993; AOTs, 1997, 1999). In order to improve degradation rates UV radiation is not used alone but combined with strong oxidants such as ozone, hydrogen peroxide, the Fenton reagent or a semiconductor photocatalyst. Processes employing UV radiation and hydrogen peroxide can be used for the remediation of water having high Color Index values originated by the presence of very low concentration of organic substances such as humic and fulvic acids (Martín *et al.*, in press). This kind of natural contamination gives to the water a persistent yellowish coloration and acts as precursor of trihalomethanes formation during conventional sanitary treatments (Callahan *et al.*, 1979).

We have studied this system [combination of low wavelength UV radiation (253.7 nm) and hydrogen peroxide] in a preliminary work (Martín *et al.*, in press) and developed a purely empirical, phenomenological reaction kinetics. Considering the difficulties in handling some of the process variables, typically the time dependence of the total absorption coefficient for example, results were analyzed in terms of the initial reaction rates.

In this work, we report the results of a kinetic study concerning water decolorization using a model for the whole reaction evolution. A simple kinetic model has been derived to describe the water decolorization process in terms of degradation rates corresponding to hydrogen peroxide and the TOC concentrations of the reacting mixture. The combination of a specially developed experimental set up and the corresponding model is a suitable tool to produce an intrinsic kinetic result, i.e., a result that is independent of the reactor configuration that can be used for scale-up purposes.

Experimental

Equipment

Gathering reliable kinetic information for scale-up purposes requires careful consideration of the geometry and characteristics of the laboratory reactor. Thus, we used a specially designed reactor. The reactor was made in such a way that considering the unfortunately low light absorption properties of hydrogen peroxide at 254 nm, almost "isoactinic" conditions can be achieved. This means that spatial variations of the radiation field for the whole reactor volume are less significant. When this is the case, when needed, an interesting simplification in the analysis of the experimental data can be carried out. In an isoactinic reactor, the "square root of the average of a given reaction variable" is almost equal to "the average of such a process variable risen to the square root power" (Brandi *et al.*, in press). When this approach is possible, and the dependence with the photon absorption rate is not linear, estimation of the reaction parameters can be simplified.

To build such a special experimental device, a flat plate reactor configuration having circular cross section was irradiated from both sides with two tubular lamps. They were placed at the focal axis of two cylindrical reflectors of parabolic cross section, made of an aluminium sheet, mirror polished, with Alzac treatment (Figure 1). This particular geometry and the employed way to irradiate the reactor, facilitate the description of the radiation field. The reactor was made of glass while the flat windows were made of quartz plates, Suprasil quality. The reactor was part of a recycling system having a storage tank, provision for maintaining a constant temperature and a recirculating pump (Figure 2). The system as a whole operated in the batch mode with very small conversion per pass in the photoreactor. Mechanical stirring in the storage tank provided good mixing conditions inside. Sampling was made in this tank. The incident flux of radiation was varied in three different levels: (i) a Philips TUV lamp (15 W), (ii) a Heraeus UV-C lamp (40 W), and (iii) a Heraeus UV-C lamp with a neutral density filter. Lamps operation was monitored with a VAW meter.

Reactor operation

Four experimental variables were investigated, three of them as a function of time exclusively:

1. the radiation absorption characteristics of the reacting mixture;
2. the hydrogen peroxide concentration (Carlo Erba RPE-ACS 30%);
3. the TOC concentration.

The Volumetric Rate of Photon Absorption (VRPA) was the fourth variable; it was varied modifying the radiation flux at the reactor windows as described above (a change that was independent of time); additional changes were due to the time changing concentrations of hydrogen peroxide (with different initial levels) and changes in all other components of the reacting mixture besides the hydrogen peroxide. Experiments were conducted at pH = 3.5 and 25ºC. The water, coming from rivers in the northern part of Argentina, was filtered (Cellulosic, Sterile, 0.45 µm) and the pH adjusted with the addition of sulfuric acid. The

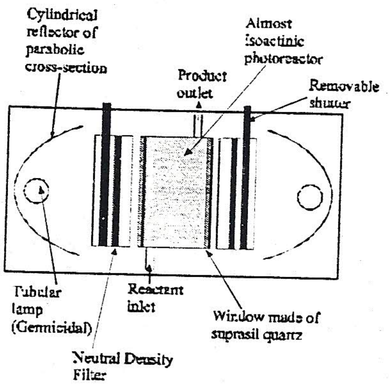

Figure 1 The reactor

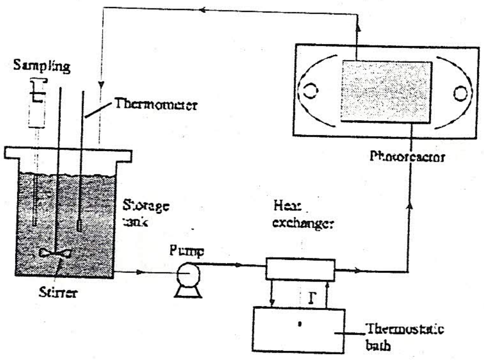

Figure 2 The reacting system

Table 1 Experimental set-up

	Parameter	Value
Reactor		
Pyrex	Diameter	4.4 cm
Suprasil	Length	4.9 cm
	Volume	74.5 cm^3
Lamps		
Heraeus UV-C	Input power	40 W
NNI 40/20	Output power at 253.7 nm	12 W
Philips TUV 15	Input power	15 W
	Output power at 253.7 nm	3.5 W
Reservoir	Volume	1000 cm^3

Table 2 Experimental program

Variable	Range
Initial total organic carbon	2.4–5.2 ppm
Initial hydrogen peroxide	50–300 ppm
Incident radiation	17.5–100%

Table 3 Reaction scheme

Reaction 1	$H_2O_2 = HP + h\nu$	→	$2\,OH^\bullet$	$k_1 = \Phi_{HP,\lambda}$
Reaction 2	$H_2O_2 = HP + OH^\bullet$	→	$HO_2< + H_2O$	k_2
Reaction 3	$H_2O_2 = HP + HO_2$[illegible]	→	$OH^\bullet + H_2O + O_2$	k_3
Reaction 4	$2\,OH^\bullet$	→	$H_2O_2 = HP$	k_4
Reaction 5	$2HO_2<$	→	$H_2O_2 = HP + O_2$	k_5
Reaction 6	$OH^\bullet + HO_2<$	→	$H_2O + O_2$	k_6
Reaction 7	$RH = TOC + OH^\bullet$	→	Prod. = P	k_7
Reaction 8	$RH = TOC + HO_2<$	→	Prod. = P	k_8

organic compound concentration in water was followed with Total Organic Carbon measurements (Shimadzu TOC-5000A) and hydrogen peroxide was analyzed with spectrophotometric methods (Allen *et al.*, 1952) (UV-vis Cary 17 D Spectrophotometer). Additional experimental information was also collected: (1) the optical characteristics (absorbance) of the reacting mixture was measured in all samples and (2) at the start and the end of representative runs, the color of the mixture was measured with the Visual Comparison Method (Eaton *et al.*, 1995). The water color index can be correlated with the total organic concentration, as it was described in Martín *et al.*, (in press).

In batch experiments, it is important to make sure that the equipment is operating under steady state conditions in all the parameters that are assumed constant; for example, lamp operation and temperatures. Before the starting of each run they were monitored during one to two hours while a shutter was interposed between the lamps and the reactor. Only when steady state conditions were achieved the desired amount of hydrogen peroxide was added to the mixture. The reaction started when both shutters were removed. Samples (30 cm^3) were taken from the tank at specific time intervals (1 h) for the different analysis. Tables 1 and 2 show more details of the reacting system and the operating conditions.

The reactor model

Four different aspects must be considered: (1) the kinetic model, (2) the radiation balance, (3) the total absorption coefficient model and (4) the mass balance.

Kinetic model

Alfano *et al.* (submitted) proposed a mechanistic approach for the UV + hydrogen peroxide (HP) degradation of 2,4-D that will be adapted here for the "hypothetical reactant" generically represented by the Total Organic Carbon (TOC) (Table 3). Values of the kinetic constants corresponding to reactions 1 to 6 are known from the literature but for pure HP. The following assumptions are made: (1) the micro steady state approximation for highly reactive intermediates (OH• and HO$_2$•) holds, (2) the reaction between the organic compounds and free radicals (OH< and HO$_2$<) is very fast and (3) for HO$_2$•, radical termination reactions with other free radicals are neglected compared with the reaction of the radical with hydrogen peroxide. Then it can be shown that:

$$R_{\mathrm{TOC},\lambda}(z,t) = -k_{\mathrm{TOC},\lambda}C_{\mathrm{TOC}}(t)\left[e^a_\lambda(z,t)\right]/2 \tag{1}$$

Where $k_{\mathrm{TOC},\lambda}$ is given by:

$$k_{\mathrm{TOC},\lambda} = \frac{(k_7k_3 + k_8k_2)\sqrt{\phi_{\mathrm{HP},\lambda}}}{\sqrt{k_4k_3^2 + k_5k_2^2 + k_6k_2k_3}} \tag{2}$$

$$R_{\mathrm{HP},\lambda}(z,t) = -k_{\mathrm{HP},1,\lambda}e^a_\lambda(z,t) - k_{\mathrm{HP},2,\lambda}C_{\mathrm{HP}}(t)\left[e^a_\lambda(z,t)\right]/2 \tag{3}$$

Where the lumped kinetic constants are given by:

$$k_{\mathrm{HP},1,\lambda} = \frac{k_2k_3k_6\phi_{\mathrm{HP},\lambda}}{k_4k_3^2 + k_5k_2^2 + k_6k_2k_3} \quad \text{and} \quad k_{\mathrm{HP},2,\lambda} = \frac{2k_2k_3\sqrt{\phi_{\mathrm{HP},\lambda}}}{\sqrt{k_4k_3^2 + k_5k_2^2 + k_6k_2k_3}} \tag{4), (5}$$

According to Luňák and Sedlák (1992) when $\left\langle e^a_\lambda \right\rangle > 1.66 \times 10^{-10}$ Einstein cm$^{-3}s^{-1}$:

$$k_{\mathrm{HP},1,\lambda}\left\langle e^a_\lambda(z,t)\right\rangle_{V_R} >> k_{\mathrm{HP},2,\lambda}C_{\mathrm{HP}}(t)\left\langle\left[e^a_\lambda(z,t)\right]/2\right\rangle_{V_R} \tag{6}$$

Our work was done under these conditions and consequently the equation can be additionally simplified. For Eqs. (1) and (3) with (6) we need the value for the local volumetric rate of photon absorption [$e^a_\lambda(x, t)$], that can be obtained from a radiation balance.

Radiation balance

The radiation distribution inside the reactor must be obtained from the solution of the radiative transfer equation:

$$\frac{dI_{\lambda,\Omega}(s,t)}{ds} + \kappa_\lambda(s,t)I_{\lambda,\Omega}(s,t) = 0 \tag{7}$$

The variable s represents the distance traveled by the radiation ray of direction Ω. Eq. (7) is valid for monochromatic light. Solving for intensities, we can obtain the spectral incident radiation (Cassano *et al.*, 1995) for the material point located at $\boldsymbol{x}$. Incident radiation is a property that accounts for radiation arriving at a given point from all directions. Then for the photochemical reaction, the activation step will be proportional to the local volumetric rate of photon absorption (LVRPA) that is given by:

$$e^a_\lambda(x,t) = \kappa_{\mathrm{HP},\lambda}\left[G_\lambda(x,t)\right] = \kappa_{\mathrm{HP},\lambda}\Omega I_{\lambda,\Omega}(x,t)\mathrm{d}\Omega \tag{8}$$

The absorption coefficient of the reactant undergoing activation is $\kappa_{\mathrm{HP},\lambda}$. In a previous paper, Alfano *et al.*, (1985) proposed a rigorous radiation field model generated by a tubular UV lamp and a cylindrical reflector of parabolic cross section. The model was experimentally verified with variable spatial position microreactors (Alfano *et al.*, 1986) and it was found that, with some restrictions in the geometry of the system, a one-dimensional model could be used. Then, according to the proposal derived in Martín *et al.*, (in press) for radiation arriving from one side and for the characteristic coordinate z, we have:

$$G_\lambda(z,t) = G_{\mathrm{W},\lambda}\exp\left[-\kappa_{\mathrm{T},\lambda}(t)z\right] \tag{9}$$

$G_{W,\lambda}$ is the Incident radiation at the inner wall of the reactor window. A similar equation can be derived from radiation arriving from the opposite side. In the case of our "isoactinic" reactor, radiation originated in lamps I and II may arrive at any point P from two sides (see Figure 1). Then,

$$G_{T,\lambda}(z,t) = G_{I,\lambda}(z,t) + G_{II,\lambda}(L_R - z,t) \tag{10}$$

If $G_{W,I,\lambda} = G_{W,II,\lambda} = G_{W,\lambda}$, substituting Eq. (9) and its equivalent for $G_{II,\lambda}$ into Eq. (10), and the resulting equation into Eq. (8), gives:

$$e^a_\lambda(z,t) = \kappa_{HP}(t) G_{W,\lambda}\left\{\exp\left[-\kappa_{T,\lambda}(t)z\right] + \exp\left[-\kappa_{T,\lambda}(t)(L_R - z)\right]\right\} \tag{11}$$

$\kappa_{T,\lambda}$ is the total absorption coefficient (colored water, reaction products and hydrogen peroxide). $G_{W,\lambda}$ is the boundary condition for radiation entrance, a value that can be calculated or be obtained with homogeneous actinometry. The same procedure will be used to confirm that the experimental boundary condition takes on the same value from both sides. With the value of the LVRPA, we can calculate the reaction rate for HP and TOC disappearances.

Total absorption coefficient model

The absorption coefficient of the reacting mixture changes with the reaction evolution. The one corresponding to HP ($\alpha_{HP,\lambda}$) is known as well as the absorption coefficient of the colored water at its initial composition $(\alpha)^0_{TOC,\lambda}$. The TOC value of demineralized water is also known (TOC $\approx 1 \times 10^{-6}$ g/cm^3). The following empirical correlation can be proposed:

$$\kappa_{T,\lambda}(t) = \alpha_{HP,\lambda} C_{HP} + \left[\alpha^0_{TOC,\lambda} + \alpha_\lambda\left(C^0_{TOC} - C_{TOC}\right)\right]\left(C_{TOC} - 1\right) \tag{12}$$

The value of a_λ can be obtained with a linear regression using the information from all experimental runs. A value of $a_\lambda = -4.23 \times 10^9$ cm^5 g^{-2} for $\lambda = 254$ nm was obtained. Depending upon the initial concentrations of HP and TOC the correlation uses a single adjustable parameter. Figure 3 shows one graphic representation of the correlation for Run 2. Knowing all elements for the reaction rate, we can write the mass balance.

Mass balance

The reaction rate derived in Eqs. (1) and (3) must be used in the mass balance for HP and TOC. Under the following assumptions: (1) isothermal operation, (2) the tank is well mixed, (3) the tank volume is larger than the reactor volume (about ten times larger is enough), (4) the conversion per pass in the photoreactor is small (differential operation), (5) the connecting lines between the tank and the reactor have a small volume and (6)

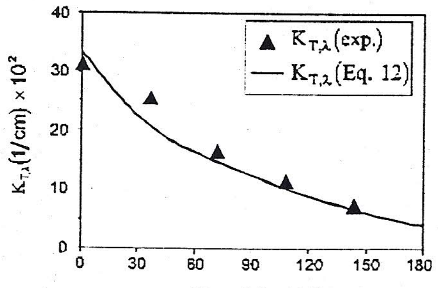

Figure 3 $\kappa_{T,\lambda}$ correlation

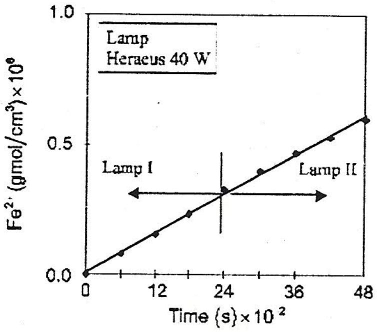

Figure 4 Experiments to evaluate $G_{W,\lambda}$

reaction occurs only inside the photoreactor, the following equation for any key species "i" (i= HP or TOC) holds (Martin *et al.*, 1996):

$$\frac{dC_i(t)}{dt} = \frac{V_R}{V_T}\left\langle R_{i,\lambda}(z,t)\right\rangle_{V_R} \tag{13}$$

V_R is the photoreactor volume while V_T is the total liquid volume of the recycling system. This equation relates changes in concentration in the tank with the reaction rate inside the photoreactor. It can be integrated with the initial condition:

$$C_i(t=0) = C_i^0 \tag{14}$$

Note that in the RHS of Eq. (13) we have the volume averaged value of the reaction rate. This follows from the evidence that sampling is taken from an averaged value of concentrations and the reaction rate is not only a function of *t* but a function of position, because the LVRPA changes with the reactor characteristic coordinate (*z*).

Experiments

Boundary conditions

Incident radiation at $z = 0$ and $z = L_R$ was evaluated with actinometry. The conventional potassium ferrioxalate actinometer was used. For the actinometer we have:

$$R_{Act,\lambda}(z,t) = -R_{Fe^{2+}}(z,t) = -\Phi_{Fe^{2+},\lambda}\, e^a_\lambda(z,t) \tag{15}$$

Using the radiation model [Eq. (11)] and the mass balance [Eq. (13)] and considering: (1) the employed actinometer has a very high absorption coefficient, (2) measuring initial rates, absorption by the reaction product can be neglected and (3) $\Phi_{Fe^{2+},\lambda}$ is known, it can be shown (Martín *et al.*, in press) that:

$$G_{W,I,\lambda} = G_{W,II,\lambda} = G_{W,\lambda} = \lim_{t\to 0} \frac{C_{Fe^{2+}} - C^0_{Fe^{2+}}}{t - t^0}\,\frac{V_T}{2A_R\Phi_{Fe^{2+},\lambda}} \tag{16}$$

This equation clearly shows that for low conversions, the absorption rate and consequently the value of the boundary condition bears a linear relationship with the change in the ferric salt composition. From Figure 4, it can be seen that this linear relationship holds and that both boundary conditions are equal. The value of $G_{W,254}$ is 14.95×10^{-9}, 5.85×10^{-9} and 2.62×10^{-9} Einstein cm^{-2} s^{-1} for the three illuminating conditions respectively.

Water decolorization

Experiments were conducted according with the program described in Table 2. We would like to model the kinetic performance employing as much as possible a mechanistically derived reaction sequence. In order to do so, equation (11) can be substituted into Eq. (1) and also into Eq. (3) with the simplification indicated by Eq. (6). These results can be incorporated into Eq. (13) with i = HP and i = TOC. Then, we must note that:

$$\left\langle C_{TOC}(z,t)\right\rangle_{V_T} = C_{TOC}(t) \tag{17}$$

$$\left\langle C_{HP}(z,t)\right\rangle_{V_T} = C_{HP}(t) \tag{18}$$

because the tank is very well mixed and the reactor operation is differential. On the other hand, the reaction rate needs the following average value:

$$\left\langle e^a_{254}(z,t)\right\rangle_{L_R} = \frac{1}{L_R}\sum_{0}^{L_R} \kappa_{HP,254}(t)G_{W,254}\left\{\exp\left[-\kappa_{T,254}(t)z\right] + \exp\left[-\kappa_{T,254}(t)(L_R - z)\right]\right\}dz \tag{19}$$

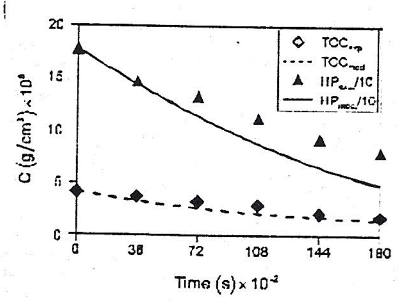

Figure 5 Run 7 – $G_{W,254} = 5.85 \times 10^{-9}$ E cm^{-2} s^{-1}

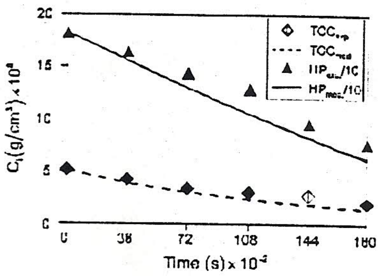

Figure 6 Run 9 – $G_{W,254} = 1.49 \times 10^{-8}$ E cm^{-2} s^{-1}

The volume average is transformed into a L_R-average because of the one-dimensional model. $\kappa_{T,254}$ is calculated with Eq. (12).

The final kinetic expressions for both the HP and TOC rates show an explicit dependence on the spatial average value of the photon absorption rate as well as the TOC and the HP concentrations. With our experimental values on concentrations and the calculated value of $\left\langle e_\lambda^a \right\rangle$ (that includes results of our correlation for the total absorption coefficient as a function of C_{HP} and C_{TOC}) we can perform a non-linear parameter estimation and obtain both kinetic constants. The result is

$$R_{TOC,\lambda}(z,t) = -15.49 C_{TOC}(t)\left[e_\lambda^a(z,t)\right]^{1/2} [=] \text{g cm}^{-3}\,\text{s}^{-1} \tag{20}$$

$$R_{HP,\lambda}(z,t) = -38.0 e_\lambda^a(z,t) [=] \text{g cm}^{-3}\,\text{s}^{-1} \tag{21}$$

Figures 5 and 6 show experimental data for two particular runs and the way that these data are represented by the kinetic model predictions under the reported experimental conditions. It can be seen that considering the implicit indefinition in the TOC concentration concept, predictions are reasonably good.

Conclusions

1. UV radiation and hydrogen peroxide can be effectively used to produce decolorization of water originated in natural contamination. The original TOC content can be reduced to almost negligible concentrations.
2. The light absorption properties of the reacting mixture changes along the reaction evolution. It can be represented with a single adjustable parameter as a function of the TOC and HP concentrations. This correlation permits the proper mathematical modeling of the radiation field inside the reactor at any reaction time.
3. A reaction scheme induced by mechanistic considerations can be used to describe the water decolorization process and the HP disappearance along the time of the reaction. Agreement between model predictions and experiments is good.
4. A new design concept, termed "almost isoactinic photoreactor" has shown to be a useful laboratory device for reaction kinetic studies with a modeling approach.

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Notation

a_λ	: Parameter in Eq. (12)	cm^5 / g^2
A_R	: Area of one reactor window	cm^2
C_i	: Concentration	g / cm^3
e^a_λ	: LVRPA	Einstein / cm^3 s
G_λ	: Spectral Incident radiation (monochromatic)	Einstein / cm^2 s
I_λ	: Spectral Radiation intensity (monochromatic)	Einstein / cm^2 s sr
k_{HP}	: Kinetic constant for HP degradation	g / Einstein
k_{TOC}	: Kinetic constant for TOC degradation	$(cm^3$ / Einstein s$)^{1/2}$
L_R	: Reactor thickness	cm
R_i	: Reaction rate	g / cm^3 s
t	: Time	s
V_R	: Photoreactor volume	cm^3
V_T	: Total volume of the system	cm^3
x	: Position in a three-dimensional coordinate system	cm
z	: Cartesian coordinate	cm
$\alpha_{HP,\lambda}$	: Specific Absorption coefficient of HP	cm^2 / g
$\alpha_{TOC,\lambda}$	: Specific Absorption coefficient of colored water	cm^2 / g
$\kappa_{HP,\lambda}$	: Absorption coefficient of HP	cm^{-1}
$\kappa_{TOC,\lambda}$	: Absorption coefficient of TOC	cm^{-1}
Ω	: Solid angle	sr

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